
بسم الله الرحمن الرحيم

الجامعة الإسلامية العالمية ماليزيا

INTERNATIONAL ISLAMIC UNIVERSITY MALAYSIA

يونيورسيتي إسلام انتارابغسا ملايسيا

Rapid Method For The Determination of Moisture Content in Biodiesel Using FTIR Spectroscopy

PRESENTATION BY

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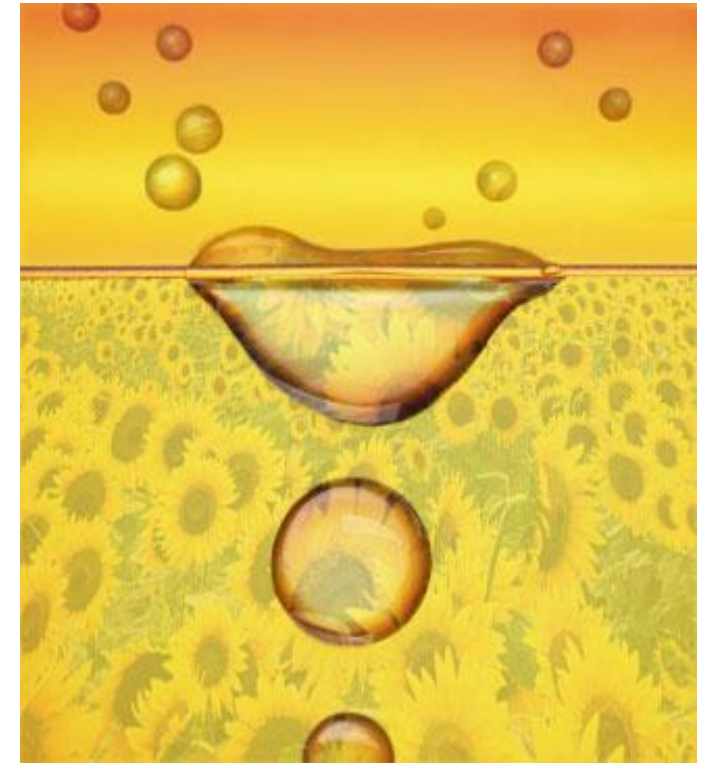
Presentation Outlines

- What is Biodiesel, Production, Sources, …
- How Moisture contaminate Biodiesel.
- Methods of Moisture determination.
- FTIR Spectroscopy
- Direct Application of FTIR
- Result
- Conclusion

Biodiesel

Biodiesel is an alternative diesel fuel made from renewable biological sources such as vegetable oils and animal fats



Sources of Biodiesel

Our sources of biodiesel here are

- *Jatropha curcas* oil,
- Sludge Palm Oil (SPO),

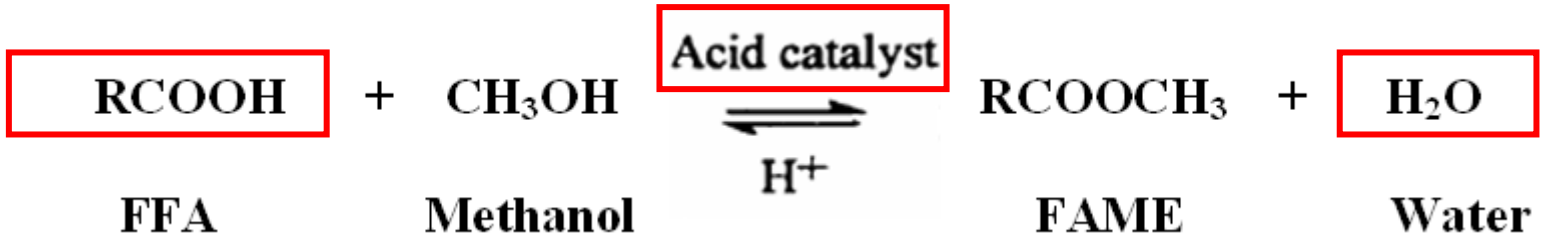
- and insects' oil.

Benefits of Biodiesel

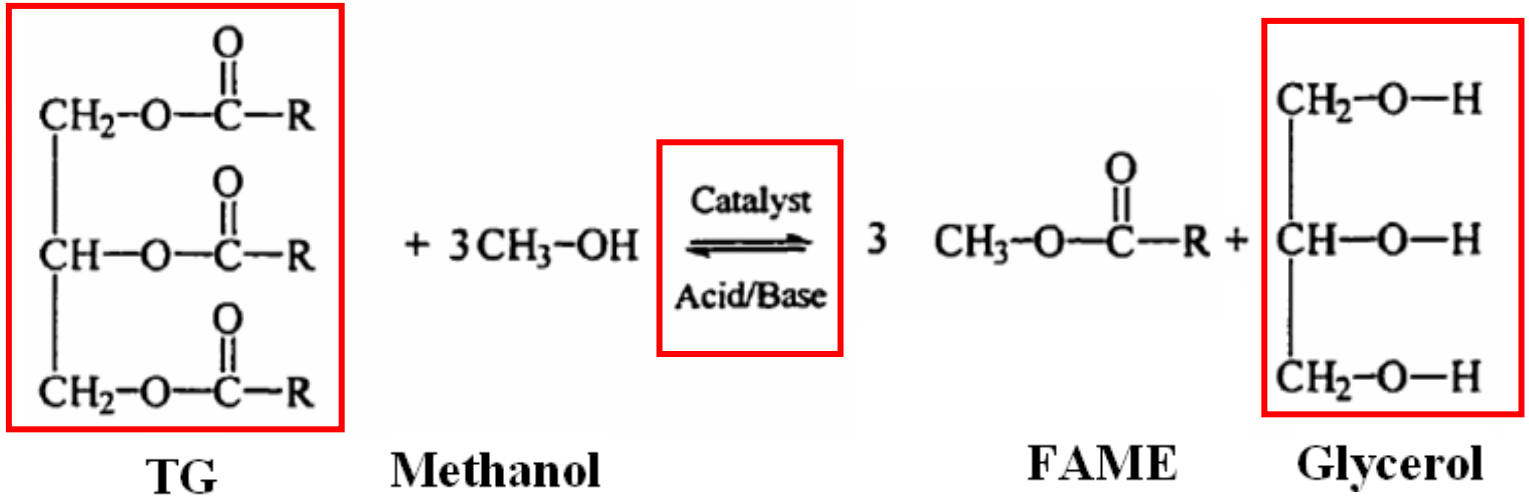
- Lower emission, lower air pollutant
- Directed used in diesel engine and blend-able with conventional diesel
- Better lubricates
- Non-toxic, renewable and biodegradable
- Such supply to reduce the dependence on petroleum

Biodiesel Production Reactions

Esterification Reaction



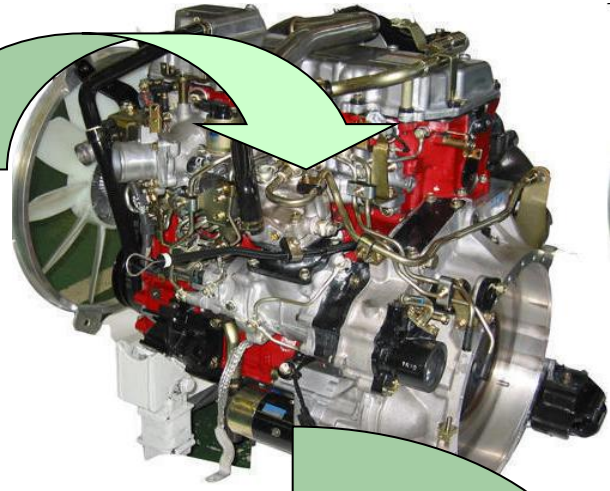
Transesterification Reaction



R = C12 to C20 straight saturated or unsaturated hydrocarbon chins

The Main Contribution

FROM SLUDGE PIT TO THE ENGINE



How does Moisture contaminate biodiesel?

- Although fats and oils and their products such as biodiesel are traded on a dry basis and packaging generally made under optimum conditions, it is virtually impossible to keep liquid, semisolid or solid materials absolutely anhydrous despite their hydrophobic nature.
- Biodiesel may contain small but problematic quantities of water. Although it is not miscible with water, it is, like ethanol, hygroscopic (absorbs water from atmospheric moisture)
- One of the reasons is the persistence of mono and diglycerides left over from an incomplete reaction. These molecules can act as an emulsifier, allowing water to mix with the biodiesel
- In addition, there may be water that is residual to processing or resulting from storage tank condensation.

Why undesirable?

- Water causes corrosion of vital fuel system components: fuel pumps, injector pumps, fuel lines, etc.
- Water may also negatively affect the heat of combustion, which leads to less power (increase fuel consumption) and may produce smoke.
- Water & microbes cause the paper element filters in the system to fail (rot), which in turn results in premature failure of the fuel pump due to ingestion of large particles (colonies).
  - Biodiesel users who have heated fuel tanks therefore face a year-round microbe problem.
- Water forms ice crystals near 0 °C. These crystals provide sites for nucleation and accelerate the gelling of the residual fuel.
- Additionally, water can cause pitting in the pistons on a diesel engine.
- You can add more …

Objectives

Our objective is to use the FTIR spectroscopy to develop a convenient rapid method for the determination of moisture content in biodiesel.

The biodiesel used here is produced from three different sources include *Jatropha curcas* oil, sludge palm oil (SPO) and insects' oil.

What is Fourier Transform Infrared (FTIR) Spectroscopy?

Fourier Transform Infrared (FTIR) Spectroscopy

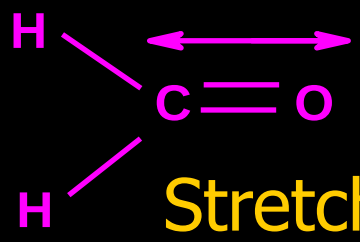
Infrared (IR) Spectrometers have been available since 1940s.

Instruments were relied on prisms as dispersive element.

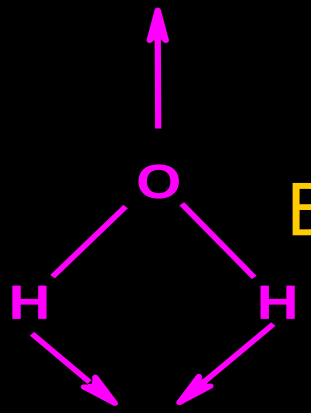
Diffraction gratings had been introduced into dispersive mechanism by the mid 50s.

The most significant advances in IR spectroscopy have come about with the introduction of Fourier Transform spectrometers.

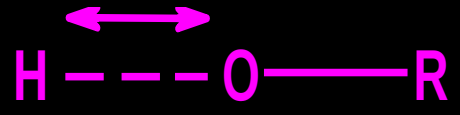
Stretching and bending vibration modes occurring in a molecule after absorbing IR energy



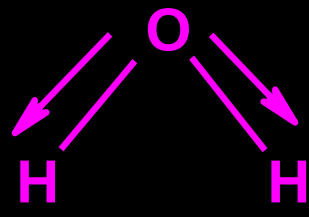
Stretching



Bending

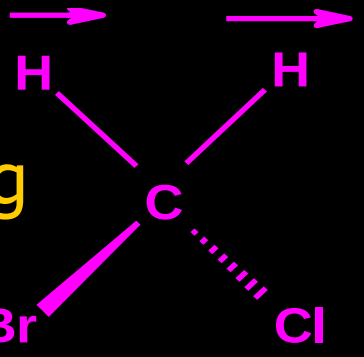


Asymmetric Stretching

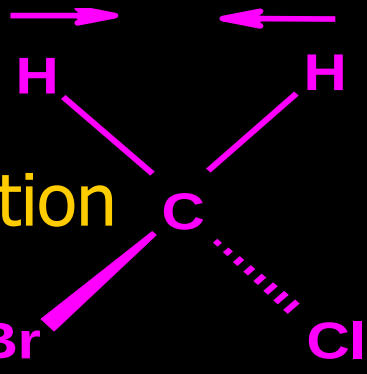


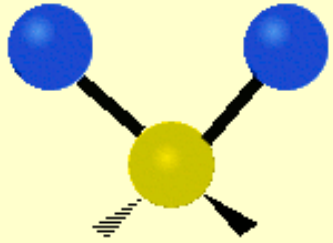
Symmetric Stretching

Rocking

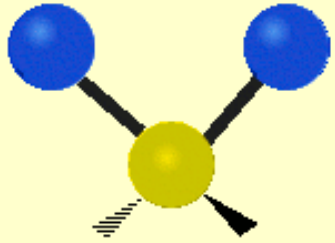


Deformation

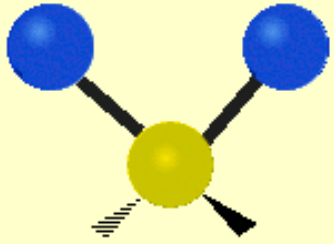




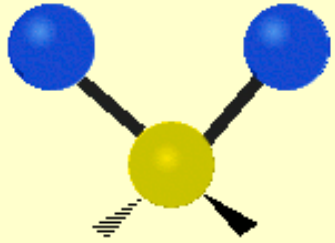
symmetrical stretching



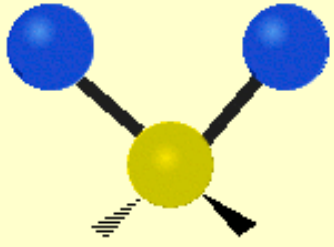
asymmetrical stretching



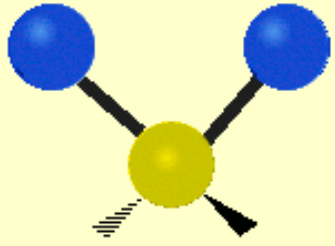
scissoring



rocking

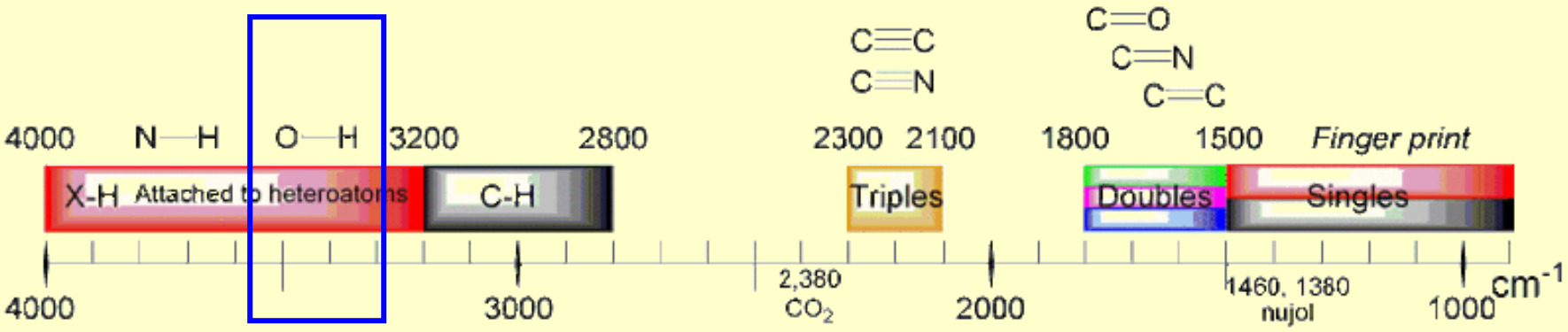


wagging

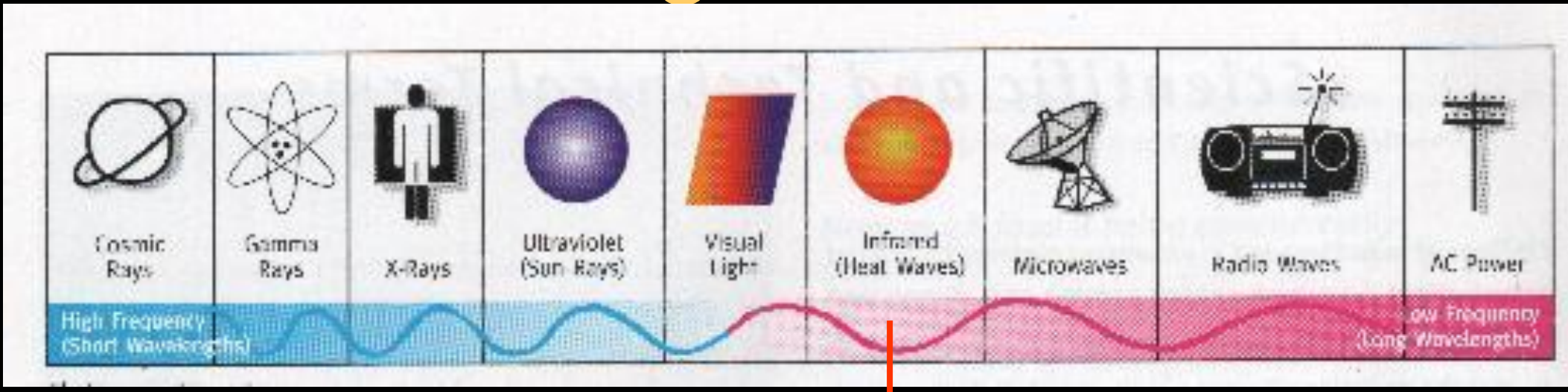


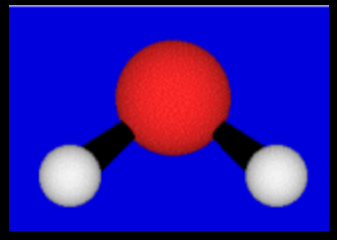
twisting

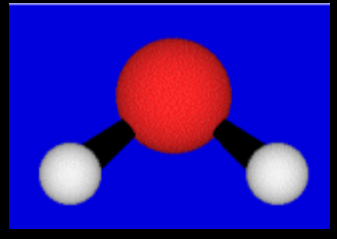
Mid IR Region

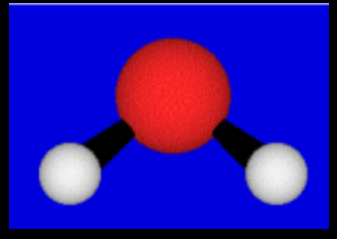


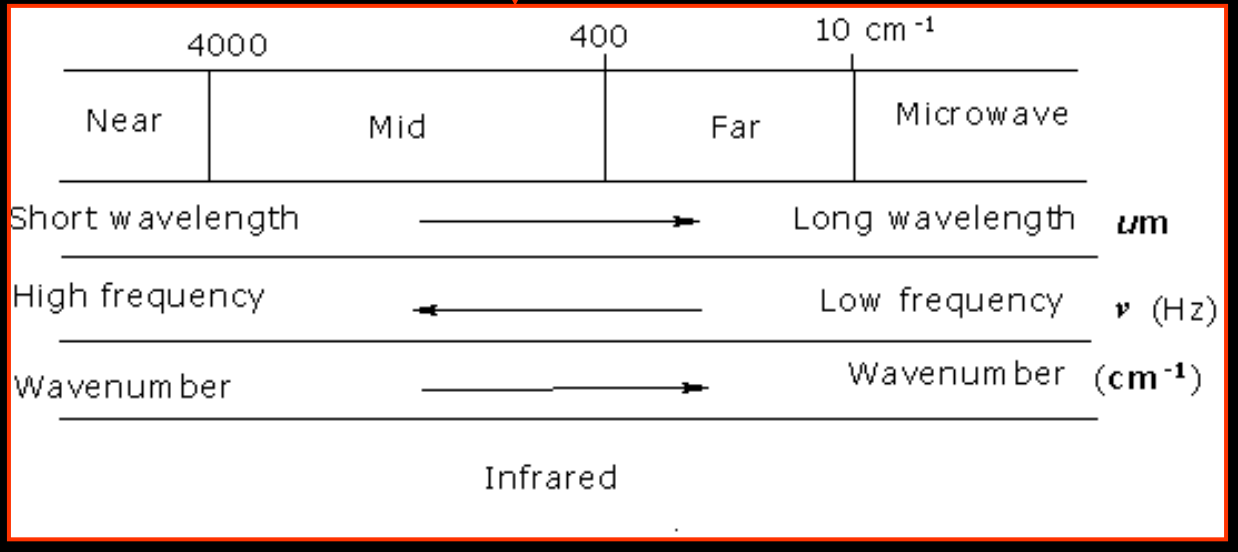
Electromagnetic Radiation



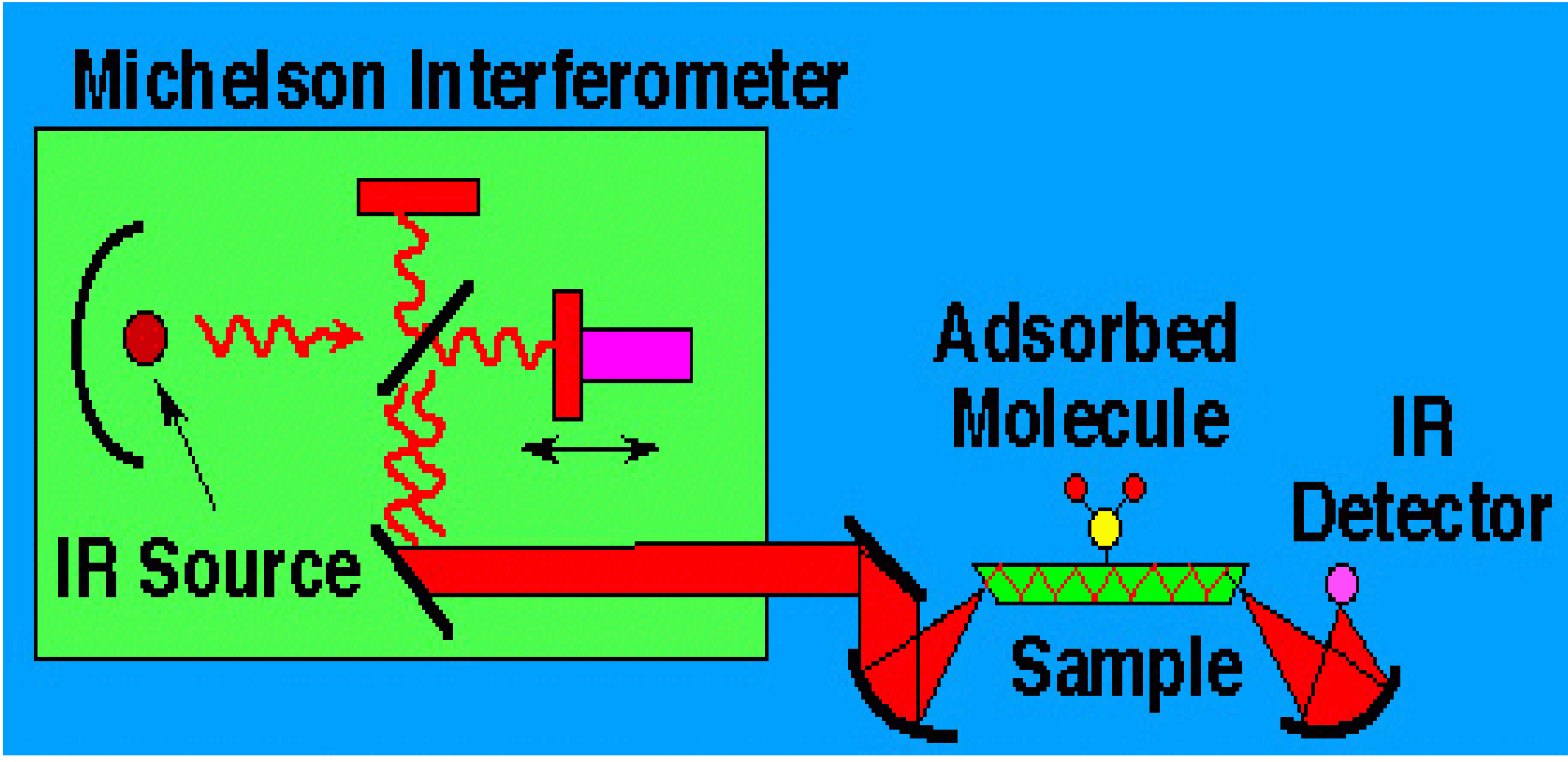






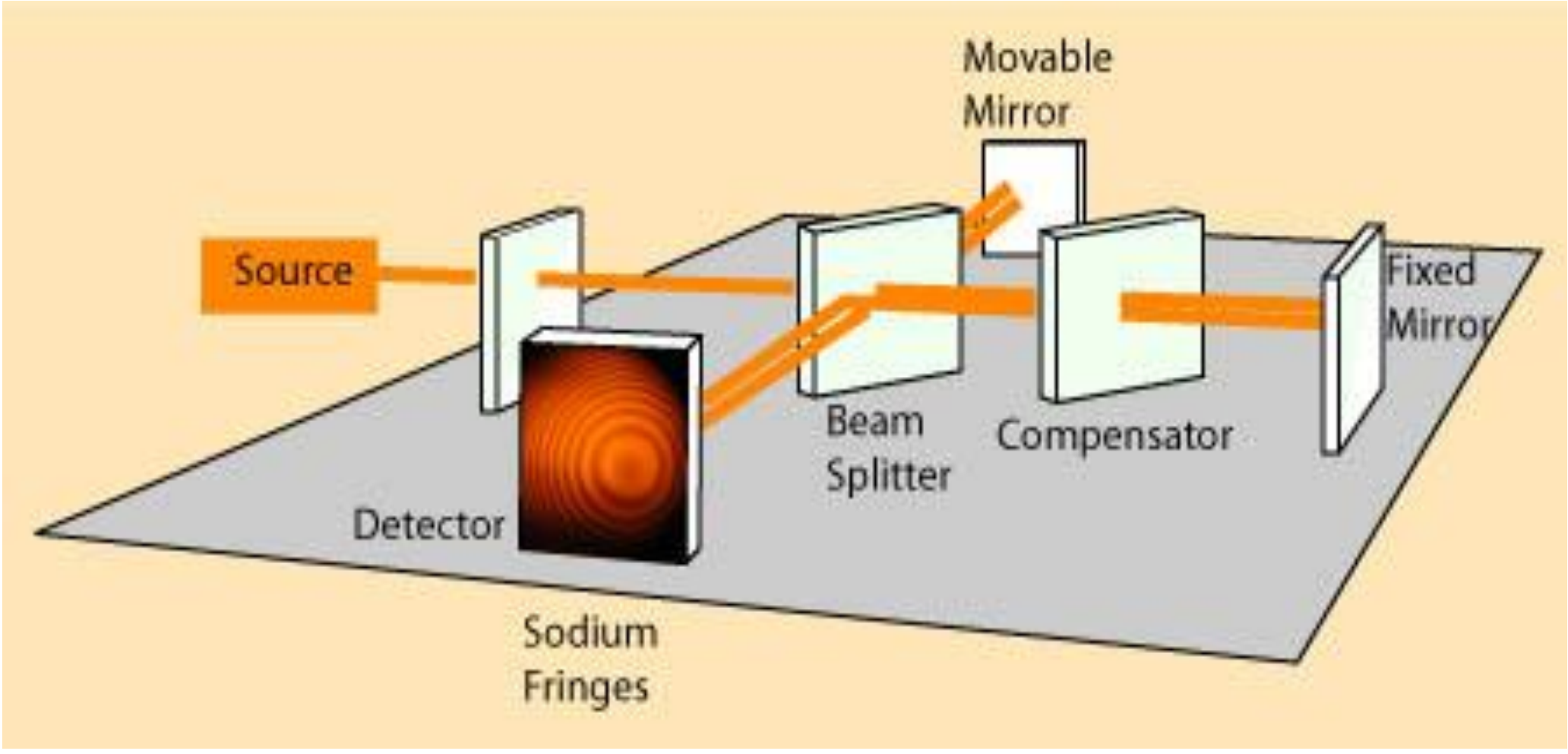


Michelson Interferometer



Used for the detection of gravitational waves, as tunable narrow band filter

Michelson Interferometer

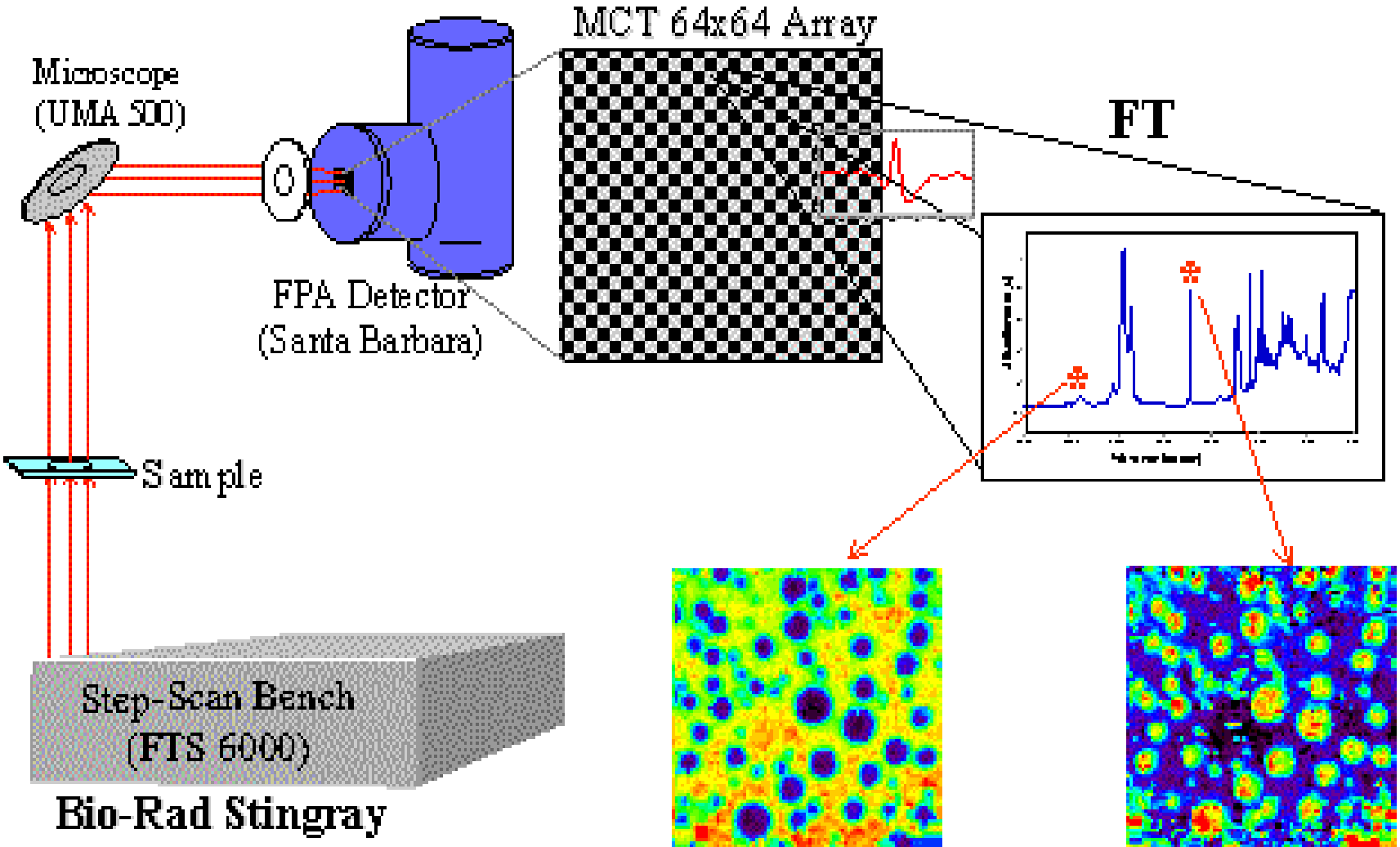


Fourier Transformation

Fluctuations in the intensity of the energy reaching the detector are digitized in real time yielding an interferogram, which contains all the information in the **time domain**.

To obtain interpretable information, the interferogram must be converted to the **frequency domain** by Fourier transformation.

Fourier Transformation



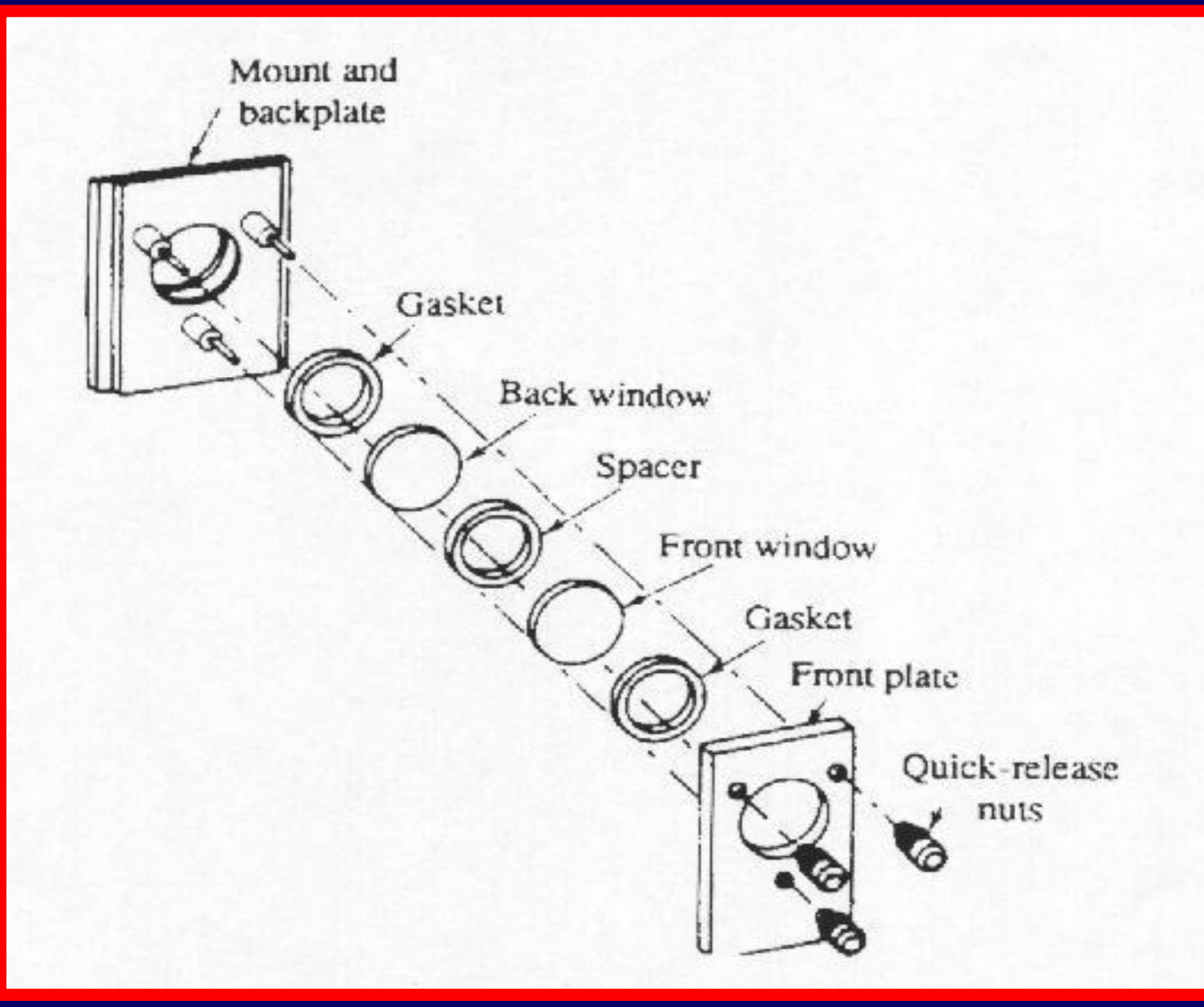
Spectral Resolution : 2 to 8 cm^{-1}
Spatial Resolution : ~ 8 μm
Collection Time : 100 s to 2 Hrs.

FTIR-Method

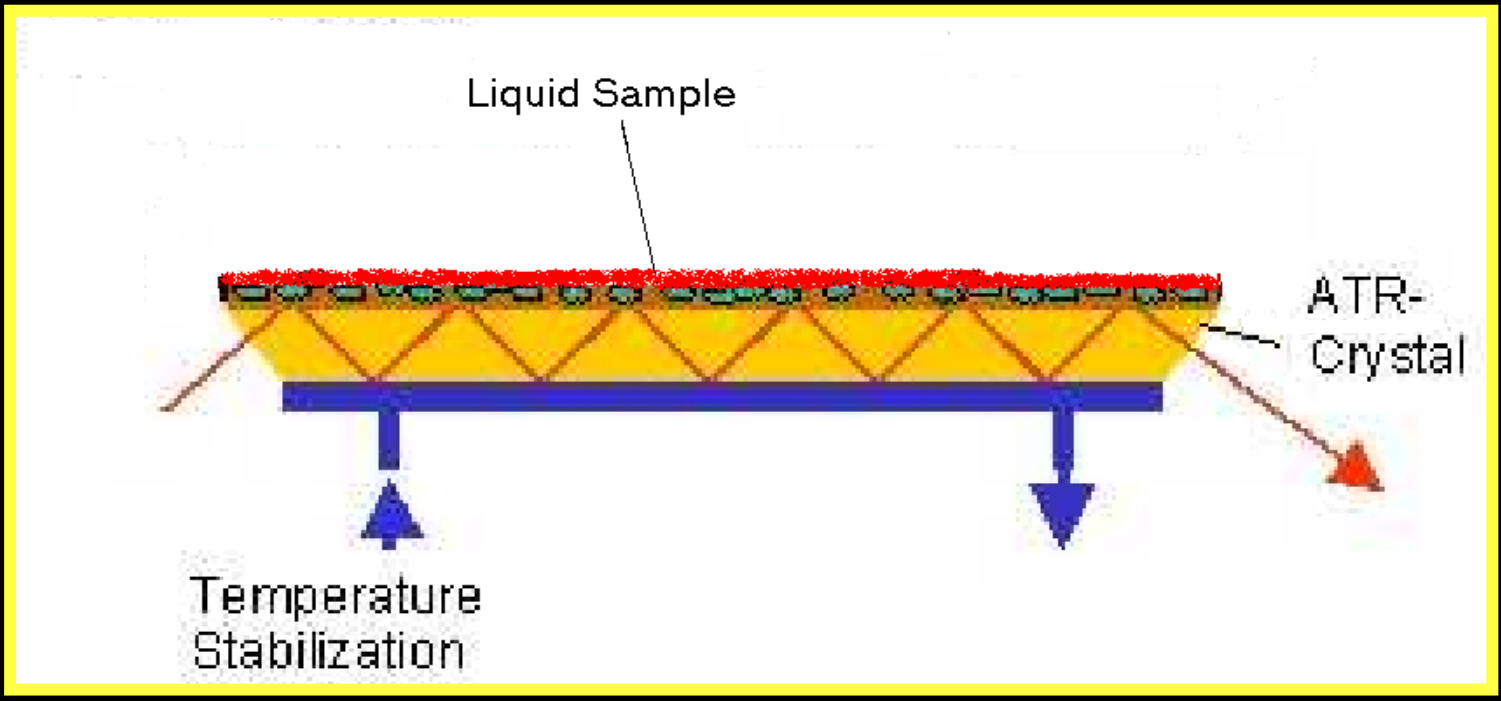
- Drops of liquid sample sandwiched between BF_2 NaCl, KBr, ZnSe, or other windows. (gas & solid samples)
- Adjust path-length.
- Initialize spectrometer, align, and collect FTIR spectra by co-adding 64 or up to 128 scans at resolution of 2 cm^{-1} over region 4000-400 cm^{-1}.

FTIR Spectrometer

FTIR Spectroscopy Transmission Cell

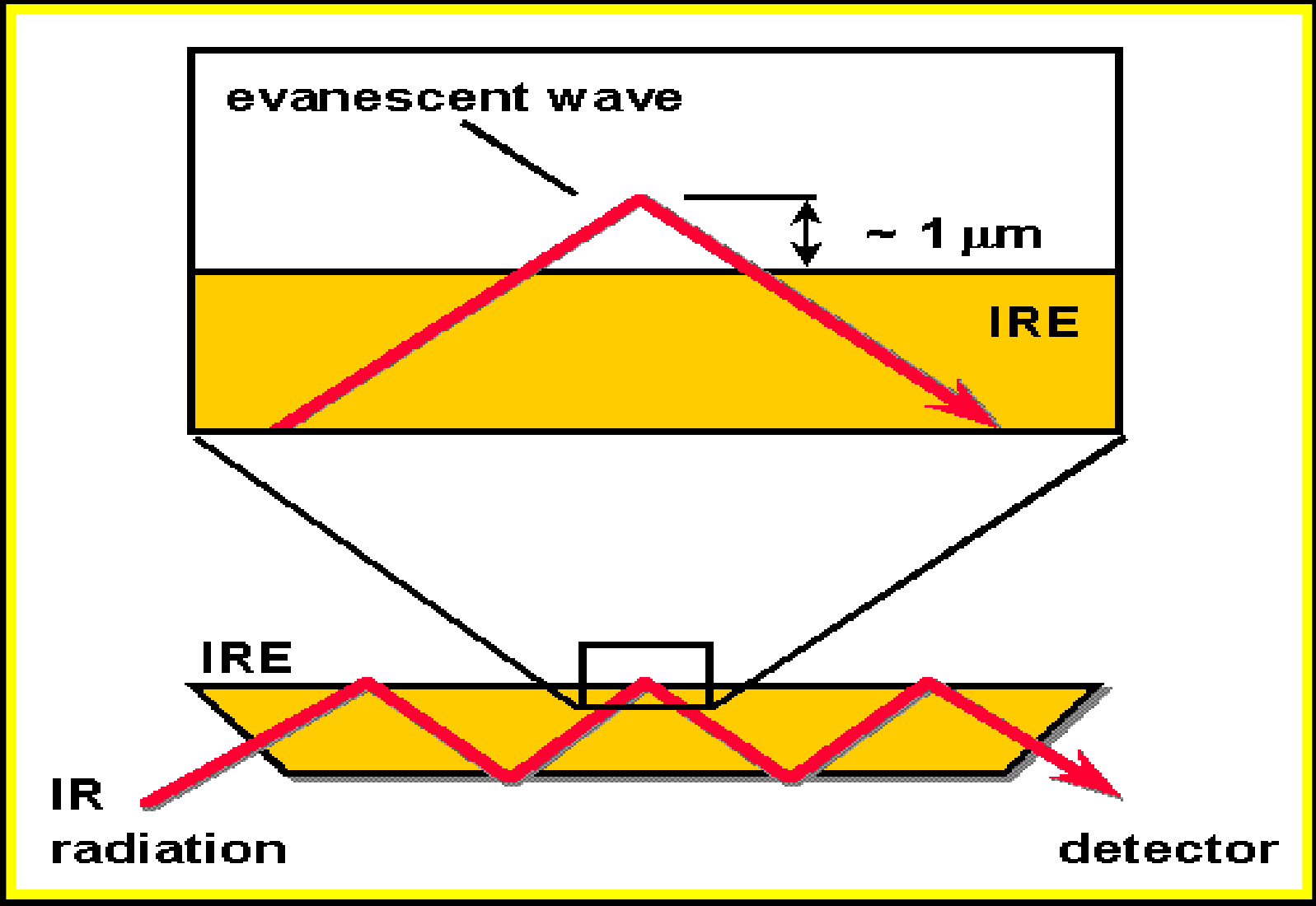


Attenuated Total Reflectance Element



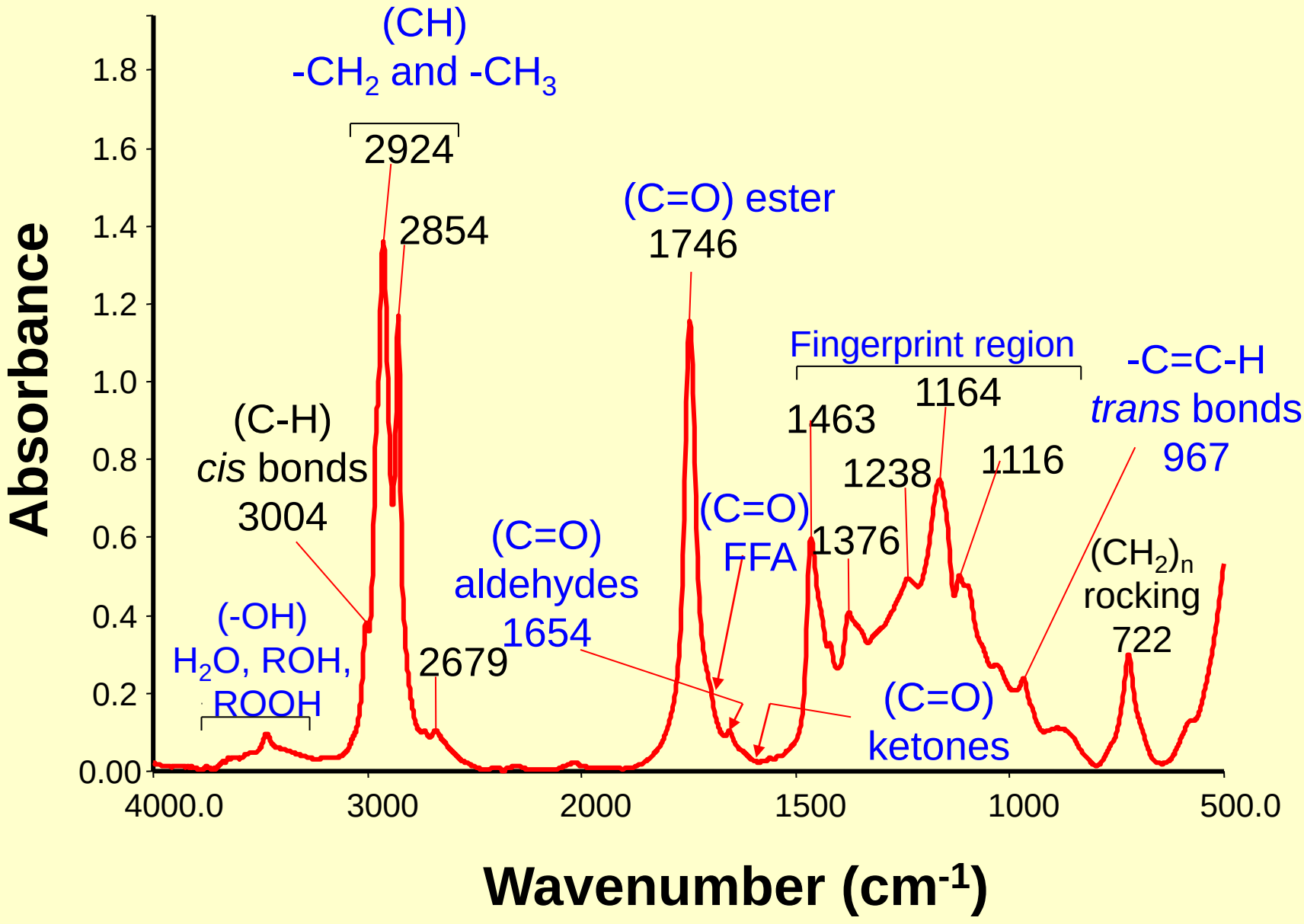
- built into a dedicated sampling station
- an 8-reflection zinc selenide (ZnSe) crystal mounted into a plate with a shallow trough for sample containment
- thoroughly cleaned by removing the previous sample with tissue and finally cleaning with distilled water and acetone

Attenuated Total Reflectance Element



Polyethylene Infrared Cards

Some of the information potentially available from an IR spectrum of oil

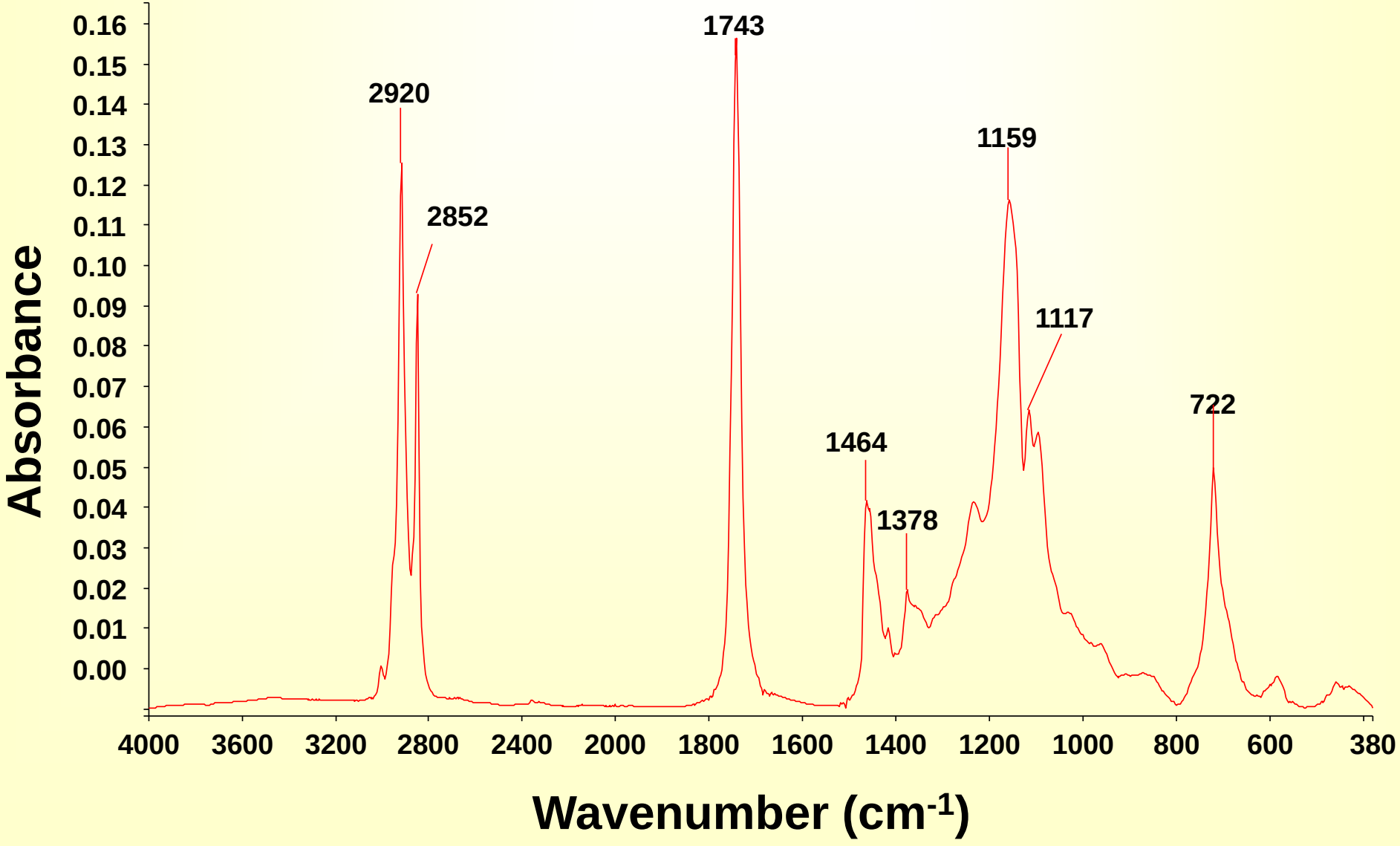


Spectral Region Select

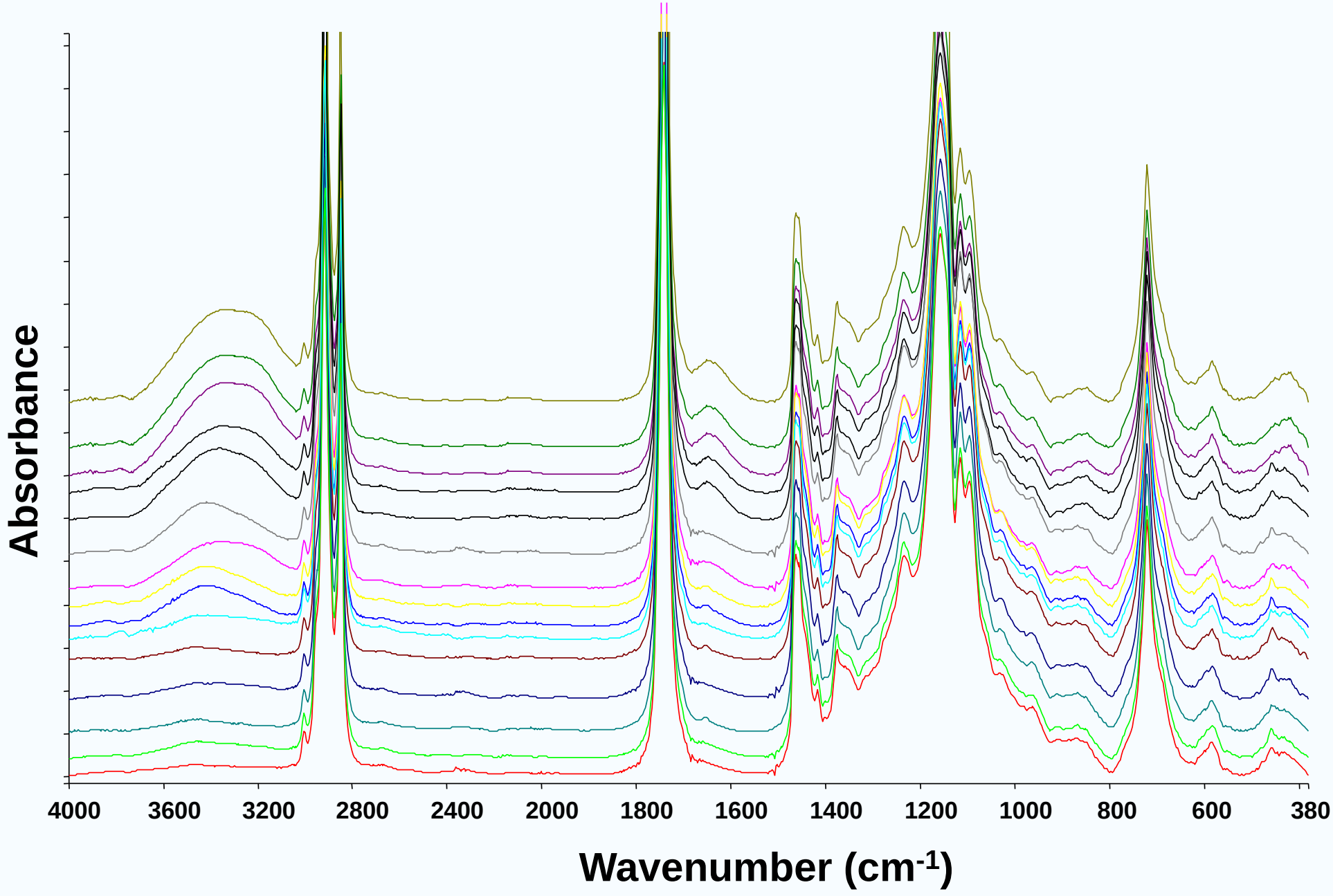
- To measure one component in a sample mixture:
  - The powerful software function recommends the best spectral regions to be measured in a quantitative method.
  - Spectral region select uses the spectra and component concentration specified in the standards to determine which regions of the spectra correlate best with component concentrations.
  - Regions **3500 – 3050 cm^{-1}** and **1680 – 1600 cm^{-1}** and partial least square (**PLS**) regression method were used to derive FTIR spectroscopic calibration model for moisture content in biodiesel samples

Results & Discussion

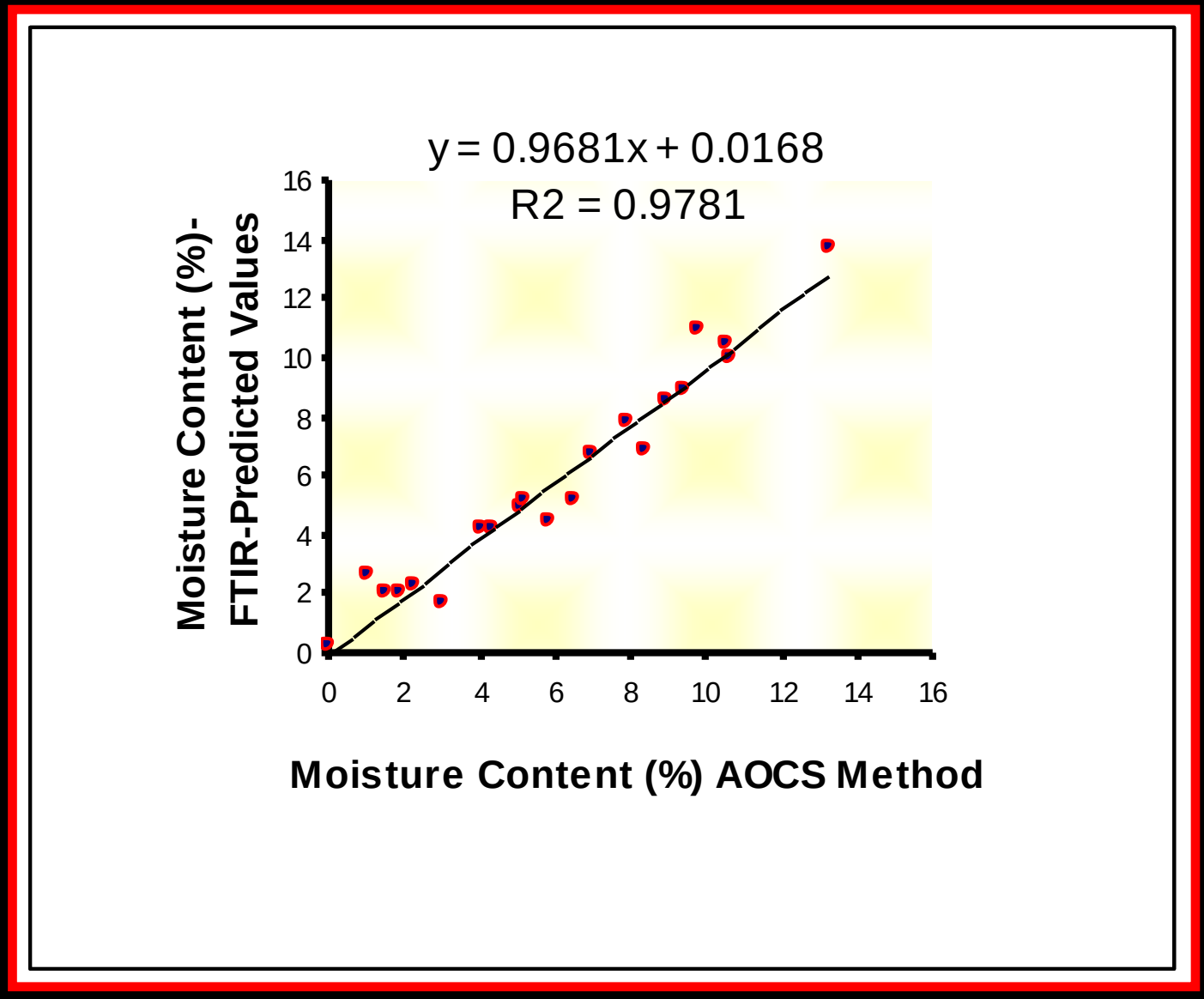
Spectrum of Pure Biodiesel



Spectra of Biodiesel with Various Moisture Content



Plot of the traditionally analyzed values of moisture from biodiesel samples versus the PLS FTIR predicted values



Validation

- Validation was carried out using leave-one-out-cross validation

Advantages

- **FTIR with advances in sample handling and data analysis tech., has greatly extended the capabilities of IR spectroscopy.**
- **FTIR spectroscopy analytical methodology for natural products was started about 25 – 30 years ago. (As a non destructive technique)**
- **As alternative to a variety wet chemical methods that are tedious time-consuming and often involve environmentally unfriendly reagents.**
- **FTIR-practical, rapid, and suitable for quality control (QC) on-line & in-line analysis.**

Conclusion

- The FTIR spectroscopy to provide a fast and accurate analysis of moisture content of biodiesel as well as crude oils.

- Multivariate calibration may also constitute an attractive alternate for fast determination of moisture content in biodiesel samples of a similar nature and composition

- The method proposed here may become an attractive alternative to Karl Fischer method and vacuum oven method, which are more tedious and time-consuming

CONCLUSION

Knowledge/Technology Advancement Contribution:

- The use of the newly introduced methods encourages many scientists' to innovate new techniques to measure the various physical and chemical properties of NP as well as to monitor quality changes during processing. Thus increased its applications both in academic research and in industry. This bode well for the wider applications of FTIR in the development of latest instrumental techniques.


الحمد لله
Thank You
شكراً لكم جزيلاً